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Status of Initial Testing of the H_2SO_4 Section of the ILS Experiment

Fred Gelbard, Robert Moore, and Edward Parma

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Fred Gelbard, Robert Moore, and Edward Parma

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Abstract

A sulfuric acid catalytic decomposer section was assembled and tested for the Integrated Laboratory Scale experiments of the Sulfur-Iodine Thermochemical Cycle. This cycle is being studied as part of the U. S. Department of Energy Nuclear Hydrogen Initiative. Tests confirmed that the 54-inch long silicon carbide bayonet could produce in excess of the design objective of 100 liters/hr of SO_2 at 2 bar. Furthermore, at 3 bar the system produced 135 liters/hr of SO_2 with only 31 mol% acid. The gas production rate was close to the theoretical maximum determined by equilibrium, which indicates that the design provides adequate catalyst contact and heat transfer. Several design improvements were also implemented to greatly minimize leakage of SO_2 out of the apparatus. The primary modifications were a separate additional enclosure within the skid enclosure, and replacement of Teflon tubing with glass-lined steel pipes.

Executive Summary

The U.S. Department of Energy (DOE), as part of the Nuclear Hydrogen Initiative (NHI), is planning to conduct laboratory-scale experiments on candidate hydrogen production technologies for production of hydrogen from nuclear energy. The first Integrated Laboratory Scale (ILS) experiment to be conducted is the Sulfur-Iodine (S-I) thermochemical cycle experiment. The ILS for S-I is a collaborative effort involving Sandia National Laboratories (SNL), General Atomics (GA), and the French Commissariat a l'Energie Atomique (CEA). The participants are each completing the construction and testing of one of the three primary reaction sections for the ILS demonstration. The construction and initial testing of all sections of the ILS experiment for the S-I process is scheduled to be completed at the end of FY07.

Sandia is responsible for the high temperature acid decomposer section for the ILS. The ILS acid decomposer incorporates the silicon carbide (SiC) bayonet heat exchanger, which integrates the vaporizer, superheater, catalytic decomposer and recuperator functions in a single, all-ceramic unit. The SiC bayonet is the central high temperature component in the acid decomposer section.

The prototype skid mounted ILS version of the decomposer was constructed and tested earlier at SNL. Based on these tests, new components were designed and built to greatly enhance the safety features and gas-handling equipment and essentially eliminate SO_2 permeation through the apparatus. The enhanced version of the decomposer was shipped to GA in late March and early April. The new design was assembled, wired, instrumented, and connected to the interface skid at GA from April to July, and chemical tests were performed in August.

This report describes the results of the initial tests of the Sandia ILS skid at GA. The new design readily achieved the desired production capacity of SO_2 for the ILS at high pressure. There was essentially no SO_2 permeation out of the apparatus. This report documents the completion of the milestone in work package SN07TC0202 due September 15, 2007 concerning integration and initial testing of the H_2SO_4 Decomposition Skid for the Sulfur-Iodine ILS Experiment.

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1 Introduction

The US Department of Energy (DOE), under the Nuclear Hydrogen Initiative (NHI), is planning to conduct laboratory-scale experiments on candidate hydrogen production technologies that are suitable for the production of hydrogen from high temperature reactors. These lab-scale experiments will provide the technical basis for the selection of the most promising nuclear hydrogen production options for the Next Generation Nuclear Plant (NGNP) in FY11.

The first Integrated Laboratory Scale (ILS) experiment to be conducted by NHI is the Sulfur-Iodine (S-I) thermochemical cycle experiment. The ILS for S-I is a collaborative effort involving Sandia National Laboratories (SNL), General Atomics (GA), and the French Commissariat a l'Energie Atomique (CEA). The participants are each completing construction and testing of one of the three reaction sections for the ILS demonstration of the S-I process.

The construction and initial testing of all sections of the ILS experiment for the S-I process is scheduled to be completed at the end of FY07. Sandia is responsible for the acid decomposer section for the Sulfur cycles and has developed a new approach to the acid decomposer that eliminates many of the high temperature corrosion and connection problems of previous efforts. The SiC bayonet heat exchanger has been designed to replace all high temperature components in the acid decomposer section. The SNL acid decomposer skid has now been connected to the French Bunsen skid and the interface skid. The SNL skid is now operating at GA and has produced SO_2 in a stand-alone mode.

This report describes the results of the initial tests of the Sandia ILS skid at GA. The new design readily achieved the desired production capacity of SO_2 for the ILS at high pressure with insignificant permeation of SO_2 out of the apparatus. This report documents the completion of the milestone in work package SN07TC0202 due September 15, 2007 concerning integration and initial testing of the H_2SO_4 Decomposition Skid for the Sulfur-Iodine ILS Experiment.

2 ILS Acid Decomposer Skid

2.1 Performance and Operational Requirements

Within the sulfuric acid decomposition section (skid 2), sulfuric acid is concentrated, heated and catalytically decomposed to sulfur dioxide, oxygen, and water. The sulfur dioxide and water produced in the process are combined with iodine and water in the Bunsen section (skid 1) to produce hydrogen iodide and sulfuric acid. The sulfuric acid is recycled back to the sulfuric acid decomposition skid. For the sulfuric acid decomposer skid to operate in the ILS, the following design objectives must be met:

- generate sulfur dioxide equivalent to a hydrogen production rate of 100 to 200 liter/hr (equivalent to 100 to 200 liter/hr hydrogen) from input acid at concentrations of 20 mol% or higher,
- maintain a decomposition temperature of 850 ºC and pressures up to nominally 3 bar,
- perform safely with redundant safety systems for containing SO_2 releases, and
- use corrosion-resistant materials of construction to allow extended operations.

The tests discussed in this report show that the skid assembled at GA for the ILS satisfies all of these objectives.

2.2 Decomposition Skid Description

A schematic of the H_2SO_4 decomposition skid developed for the ILS experiments at GA is shown in Figure 2.1. Dilute acid from the Bunsen skid enters the concentrator where the acid is concentrated from 20 mol% to about 40 mol%. The concentrated acid is collected into the Concentrated Acid Tank, and the water vapor is condensed and collected in Water Tank 1. The acid is then combined with unreacted acid from the bayonet and introduced into the base of the bayonet. In the bayonet the acid is heated to 850 °C and partially decomposed. The gaseous decomposition products of SO_2, O_2, and H_2O flow out the inner channel and most of the heat content is recuperated to the incoming acid. Undecomposed acid is condensed, collected in the Recycle Acid Tank, and recycled back to the bayonet. The gas stream is then cooled to condense liquid water into Water Tank 2. A gas stream consisting of SO_2 and O_2, and a liquid water stream from Water Tanks 1 and 2 are the products of the H_2SO_4 decomposition skid. These two streams are sent to the Bunsen skid.

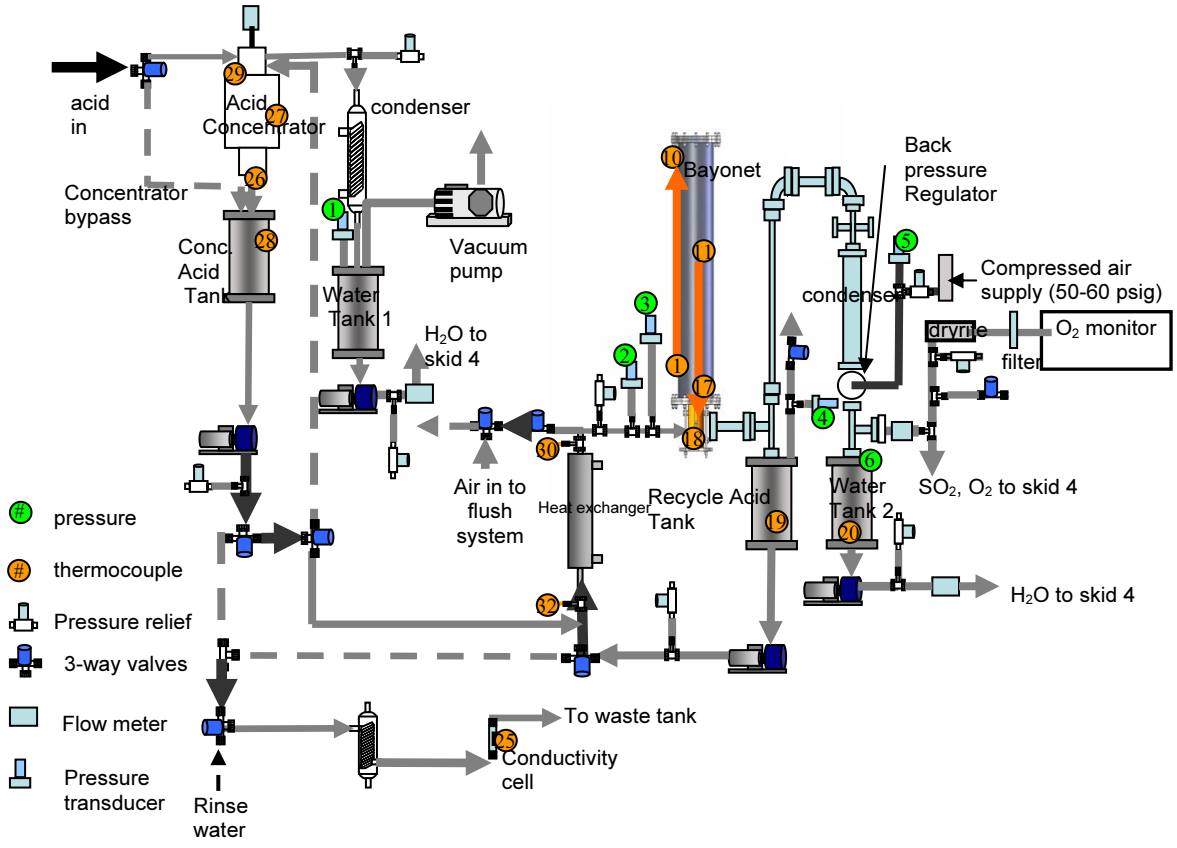

Figure 2.1 Schematic layout of the H_2SO_4 decomposition section skid. The orange and green shaded circles correspond to the locations of thermocouples and pressure transducers, respectively. The shapes of the bayonet, concentrator, water tank 1, water tank 2, recycle acid tank, and the concentrated acid tank in this Figure are drawn to be similar to the actual units shown in Figures 2.3 and 2.4.

The entire H_2SO_4 decomposition skid is contained within a Lexan enclosure that has a 4 ft by 4 ft base, and is 8 ft high. The two access sides of the skid are shown in Figures 2.2 and 2.3, respectively. The outer enclosure is connected to the GA ventilation system, which continuously draws 800 cfm from within the enclosure through a charcoal filter before releasing the gas to the atmosphere.

Within the outer enclosure the components are segregated into two different areas of the skid; those containing liquid acid solutions shown in Figure 2.2, and those containing SO_2 shown in Figure 2.3. Previous tests at SNL showed that SO_2 is highly permeable through Teflon and Viton. To minimize the possible releases of SO_2 two design features were added to the system. First, glass-lined steel was used on the outlet side of the bayonet to the product outlet to minimize the release of SO_2 from the unit itself. Second, an inner Lexan enclosure was constructed within the primary outer enclosure to confine any SO_2 permeating

from the system. As shown in Figure 2.3, the bayonet and other SO_2 containing components were arranged within an inner confinement enclosure. Other components that contain only acid solutions are arranged within the primary outer skid enclosure. Gas leakage from the apparatus within the inner enclosure is contained. The inner enclosure has a separate activated charcoal filter system to absorb small gas leaks. A blower can be remotely operated to vent the inner enclosure to the outer enclosure, which then is vented to the GA ventilation system. Both the inner enclosure volume and the primary outer enclosure volume contain SO_2 monitoring systems that allow for continuous readout both at the skid and at the control panel. A third SO_2 monitor is mounted on the outside of the skid to detect leakage outside the enclosures. Furthermore, two handheld SO_2 monitors are used when running an experiment or servicing the skid.

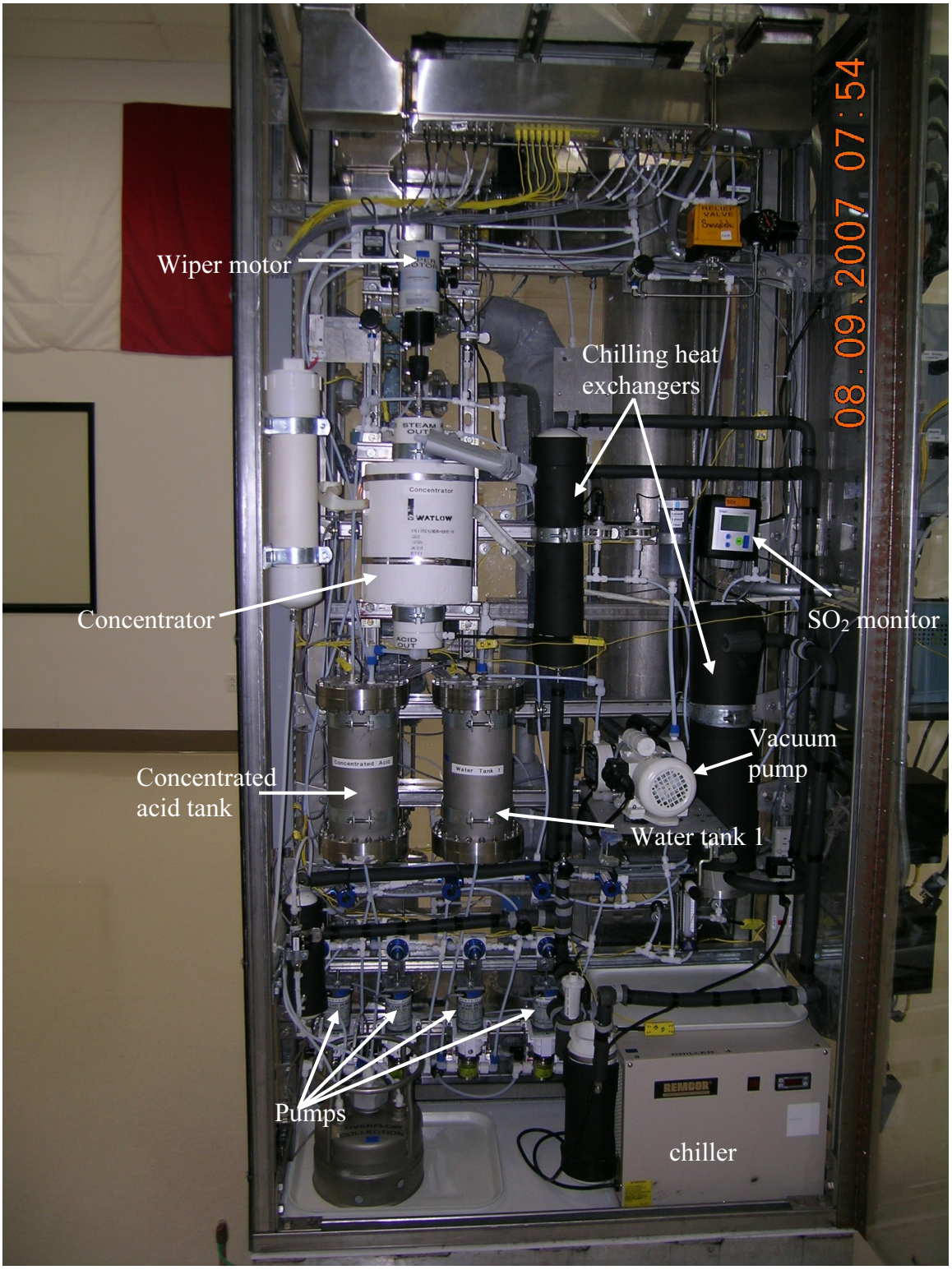

Figure 2.2 The liquid acid side of the acid decomposition skid assembled at GA.

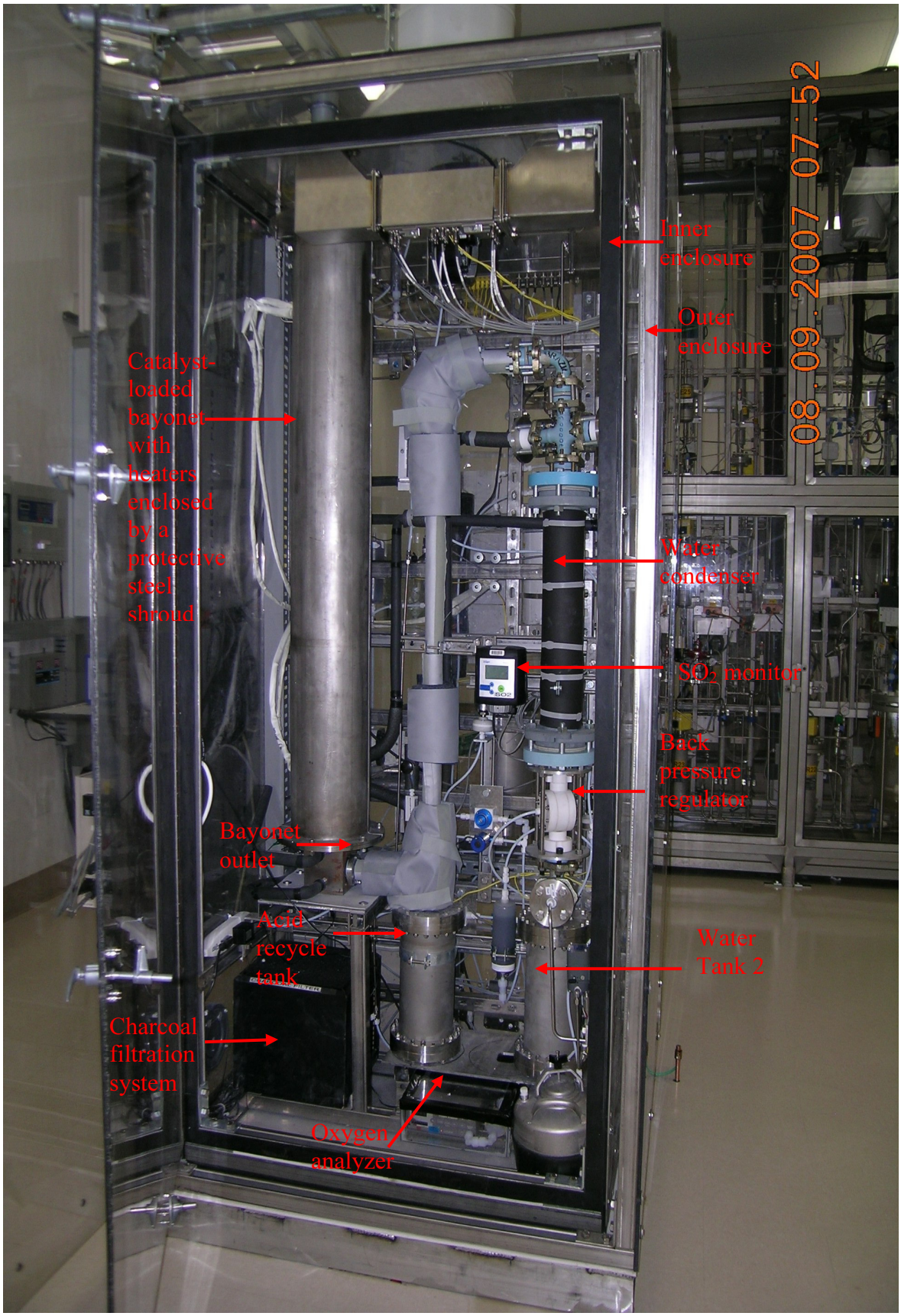

Figure 2.3 The SO_2 side of the acid decomposition skid assembled at GA.

2.2.1 **Acid Concentration**

Sulfuric acid enters the skid at approximately 20 mol%. Acid heating and acid concentration are accomplished in an electrically heated heat exchanger constructed of a SiC pipe with Teflon end caps. A schematic diagram of the concentrator in Figure 2.2 is given below in Figure 2.4.

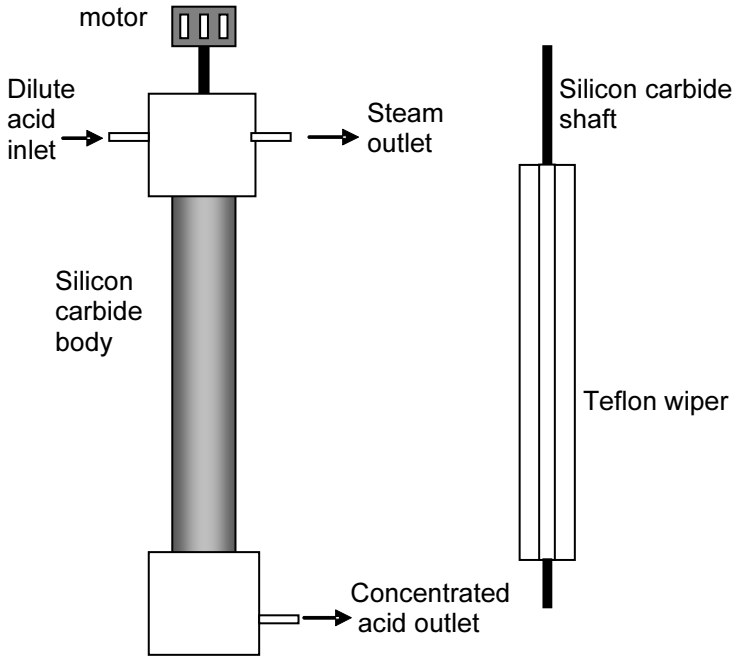

Figure 2.4 Schematic of concentrator, excluding the surrounding electric heater.

The concentrator is a wiped-film type unit with a rotating inner Teflon wiper to maintain liquid contact with the heated walls. The concentrator is under vacuum which removes water vapor and concentrates the acid to approximately 40 mol%. Dilute acid enters the top of the concentrator and travels downward. The wiper, driven by a laboratory stirrer, increases contract of the liquid with the heated SiC wall and increases the path length of the acid through the apparatus. For optimum efficiency, the concentration process is performed under sub-atmospheric pressure of approximately 1.0 psia. By lowering the pressure and therefore the boiling temperature of the acid, the energy required to heat the acid to its boiling point is decreased and input heat is mainly used for the heat of vaporization of the water from the dilute acid solution. Heat is supplied to the concentrator using a ceramic heater. Water is removed from the top of the apparatus as steam which is cooled to liquid water in a water-cooled condenser.

The water is collected in a stainless-steel pressure vessel labeled Water Tank 1 in Figures 2.1 and 2.2. A vacuum pump connected to the tank is used to maintain vacuum in the system including the acid concentrator pressure.

2.2.2 **Bayonet Acid Decomposer**

The bayonet is shown schematically and as disassembled components in Figure 2.5. The operations of the bayonet have been described in previous reports (Moore et al., 2007; Gelbard et al. 2007). The basic operating parameters for the unit are the acid flow rate, the inlet acid temperature, and the maximum temperature. The bayonet is heated using electrical heaters controlled to a maximum temperature at the outside top portion of the bayonet. The products of acid decomposition are sulfur dioxide, oxygen and water vapor. These are separated from undecomposed acid in the lower section of the inner SiC tube.

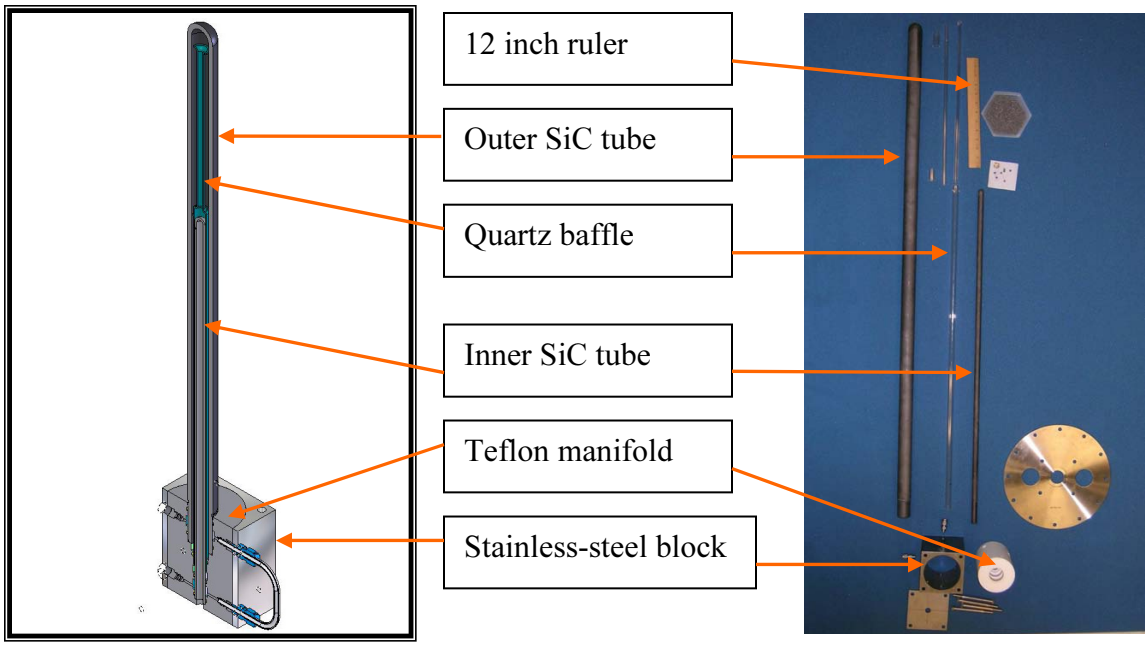

Figure 2.5 The SiC bayonet decomposer shown schematically on the left and the actual hardware on the right.

2.2.3 ILS Decomposition Skid Control Description

The control system and control panel to operate the acid decomposition skid have been assembled at GA. Control of the skid is maintained by a computer server located in the control room and an Ethernet connection to an Allen Bradley control system located on a control panel outside of the skid enclosure. Hardwired manual safety shutdown systems and purge-valve controls are also located in the control room. A second, hardwired, manual, safety shutdown is located on the skid control panel. A third, automatic, hardwired, pressure shutdown system is incorporated into the control panel. The control system and methodology for the acid decomposition skid was developed to be similar to the system used by GA for the HI decomposition skid.

Diagnostic information is displayed and evaluated including valve positions, tank levels, temperatures, pressures, flow rates, SO_2 levels, and other transducer diagnostic measurements. Alarm and shutdown conditions are also displayed. Using the mouse and keyboard, valves can be opened and closed, pumps turned on/off and speeds changed, heater controller temperatures changed, and equipment turned on/off.

Control of the acid decomposition skid is relatively straightforward. The system has been found to be easily controlled, started up, and shut down. The electrical heating of the system (concentrator and bayonet) does not have to be rigorously controlled with process flow feedback. The heaters can be set and the controller will work with or without fluid flow.

2.2.4 Inter-Skid Connections

For integrated testing, the sulfuric acid decomposition section (skid 2) must be connected to interface skid (skid 4) and the Bunsen section (skid 1). A diagram indicating the plumbing connecting the skid is shown in Figure 2.6, and the actual components on the interface skid are shown in Figure 2.7. Dilute sulfuric acid is received at the interface skid from the Bunsen section. The acid contains small amounts of iodine that are removed by passing it through a cold trap followed by an activated carbon filter. The acid is then stored in Teflon-lined stainless-steel tanks for use by the sulfuric acid decomposer section. The dilute acid is pumped to section 2 where it is concentrated and subsequently decomposed. Water from the concentration and acid decomposition processes is pumped through Teflon-lined stainless-steel tubing to the interface skid for use in the Bunsen skid. The SO_2 and O_2 are pumped through 0.5” ID Hastelloy tubing either to the Bunsen skid or the scrubber located on the interface skid. Unlike all other chemicals used in the S-I process, SO_2 is not stored on the interface skid. For flushing out skid 2, a Teflon-lined stainless-steel vent line connects skid 2 to the scrubber on the interface skid.

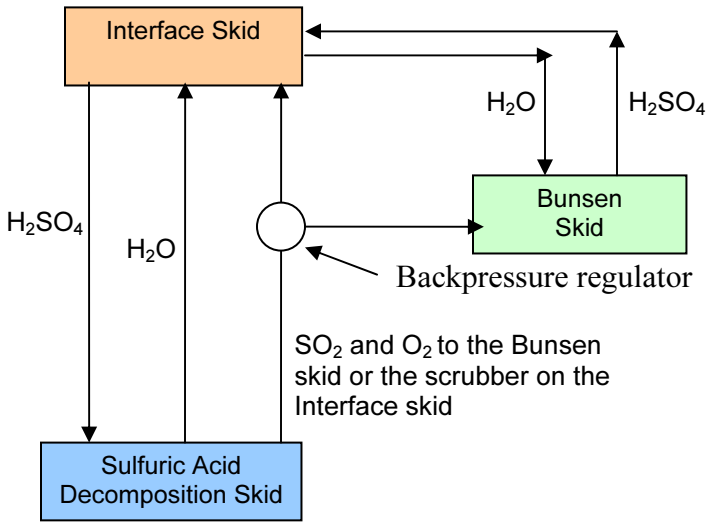

Figure 2.6 Flow streams between the Sandia acid decomposition skid and the French Bunsen skid.

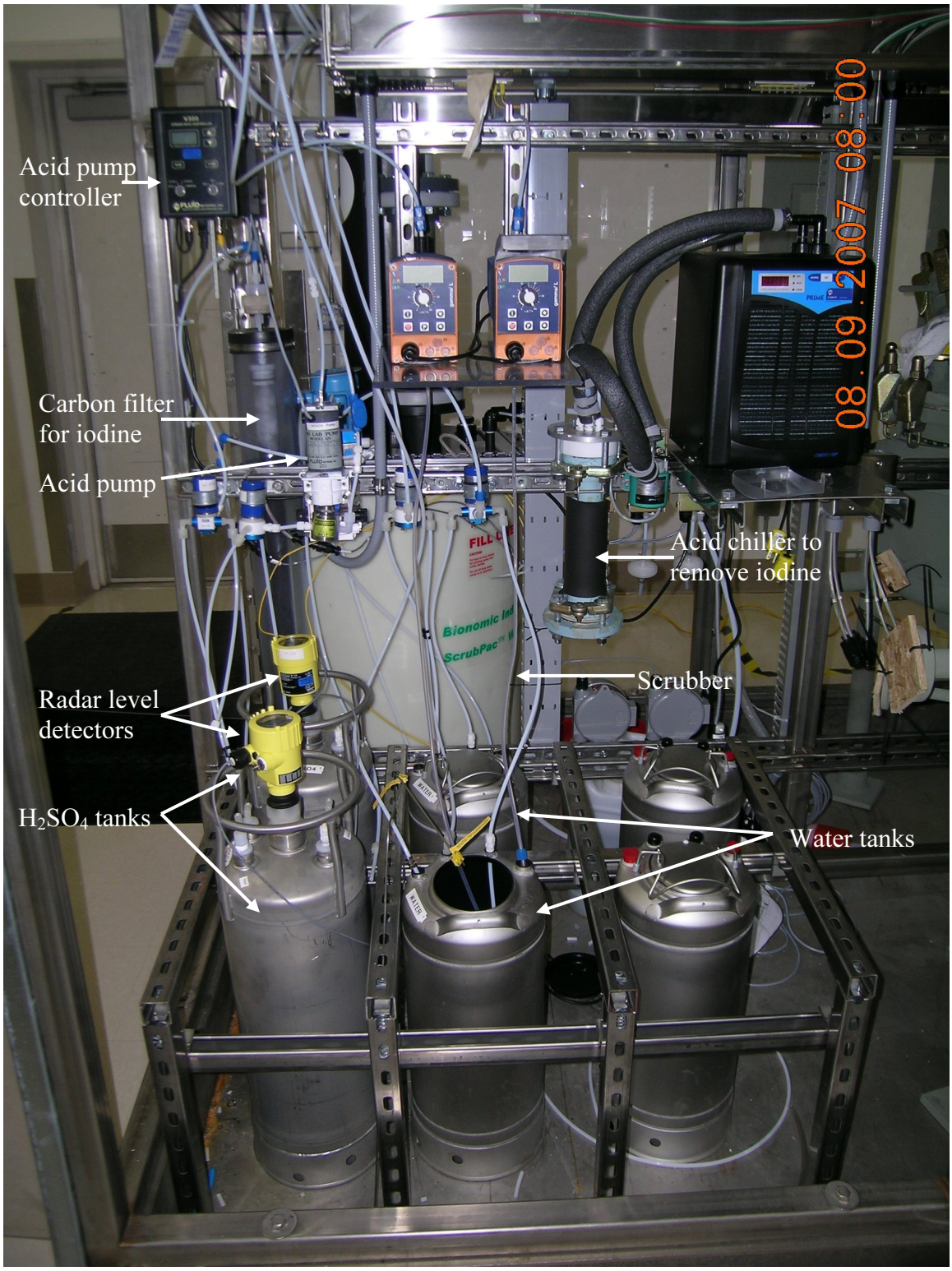

Figure 2.7 Interface skid components used to store sulfuric acid and water that are exchanged between the sulfuric acid section (skid 2) and the Bunsen section (skid 1).

3 Experimental Tests and Results

3.1 Concentrator Acid Tests

The acid concentrator has been tested to confirm operation and performance. Dilute acid contained in the Teflon-lined, stainless-steel vessel located on the interface skid was pumped to the H_2SO_4 decomposition skid and into the acid concentrator. Two tests were performed with the concentrator operating with an input acid flow rate of 48 and 50 ml/min. The test temperatures and pressures were 175 ºC at 1.0 psia and 190 ºC at 3.0 psia, respectively. Acid output from the concentrator was collected in the concentrated-acid tank on the skid. The output acid concentration was determined by withdrawing a sample of the acid and measuring the density. The concentrated acid is the feedstock for the acid decomposer. Steam exiting the top of the concentrator was condensed and collected in the water-collection tank located on the skid.

The results of these two tests are given in Table 3.1. Dilute acid was concentrated to 38 and 45 mol%, the values prescribed in the design criteria. In the second test, a small vacuum leak developed resulting in operation of the apparatus at 3.0 psia instead of the desired value of 1.0 psia. By increasing the operating temperature from 175 ºC to 190 ºC an output acid concentration of 45 mol% was achieved. The vacuum leak was caused by a faulty seal and this problem has been corrected and is not expected to create future problems.

Table 3.1 Testing results for the sulfuric acid wiped film concentrator.

Input acid concentration (mol%)	Flow rate of acid input (ml/min)	Operating temperature of outside wall (ºC)	Operating pressure (psia)	Output acid Concentration (mol%)
20	48	175	1.0	38
19	50	190	3.0	45

3.2 Bayonet Acid Decomposition Tests

The two primary objectives of the initial tests were to demonstrate SO_2 production capacity of at least 100 liters/hr, and pressurized operations up to 3 bar (45 psia). To achieve these objectives, the flow rate and back pressure were increased in sequence as given in Table 3.2. For all phases of the test, the catalyst region of the bayonet was maintained at 850 °C and the acid input concentration was 31 mol%.

Table 3.2 Sequence of acid flow rate and back pressure

Sequence Index	Time Interval	Acid flow rate (mol/hr)	Back Pressure (bar)	Back Pressure (psia)
1	12:00 - 12:12	3.6	1	15
2	12:12 - 12:22	7.1	1	15
3	12:22 - 12:26	7.1	1.5	23
4	12:26 - 12:30	7.1	2	30
5	12:30 - 12:42	7.1	3	45

Acid injection began slowly to avoid any thermal shock to the system. Initially the acid flow rate was maintained at 3.6 moles/hr and gas production started at about 12:00 noon. For this small flow rate the inlet pressure given in Figure 3.1 was about 18-19 psia, and no back pressure was applied. Thus the outlet pressure was at ambient, nominally 15 psia. The inlet pressure measurements tended to be high by about 1 - 2 psia. Thus the pressure drop across the bayonet was only about 2 psia. At 12:12 PM the second sequence started and the acid flow rate was increased to 7.1 moles acid/hr. This flow rate was held constant for the rest of the experiment as the pressure was varied.

As given in Table 3.2, the pressure was incremented in three steps by increasing the back pressure from ambient to 1.5, 2, and 3 bar, at 12:22 PM, 12:26 PM, and 12:30 PM, respectively. Because there was only about a 2 psia pressure drop across the bayonet, the inlet pressure given in Figure 3.1 is essentially also the pressure in the catalyst region. Notice that the system attained the set pressure typically within a minute. We started to gradually reduce the back pressure at 12:42 PM to ambient by 12:50 PM, and then the test was terminated.

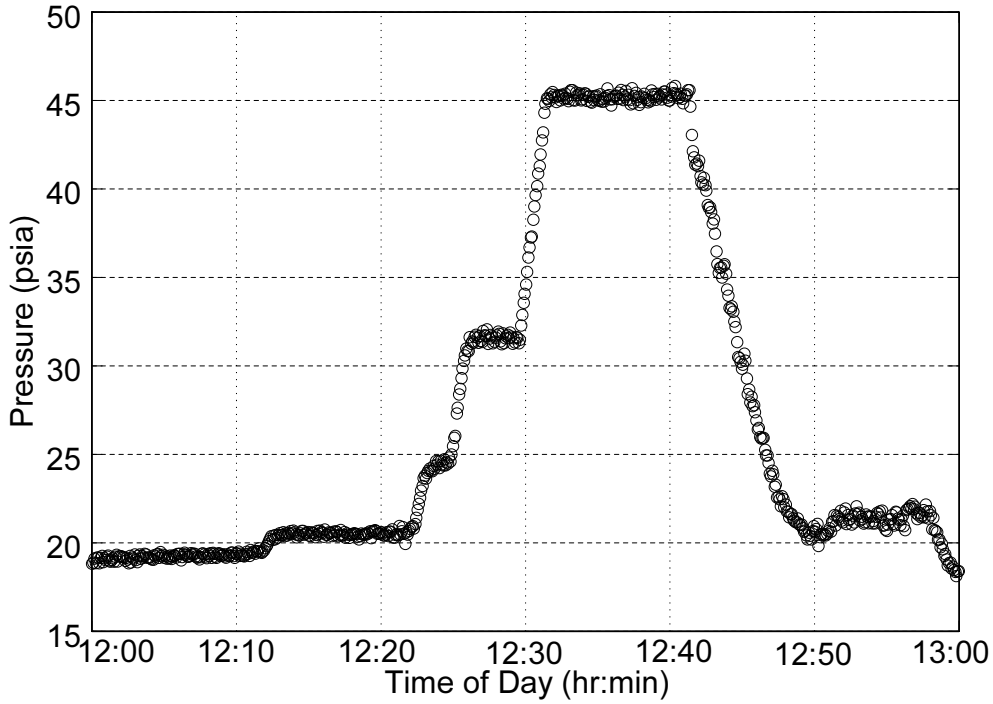

Figure 3.1 Inlet pressure to the bayonet.

The production rate of SO_2 is plotted in Figure 3.2. The acid flow rate into the bayonet from 12:00 PM to 12:12 PM was only 3.6 mol/hr, and the resulting SO_2 production rate attained about 75 l/hr. When the acid flow rate was increased to 7.1 mol/hr at 12:12 PM, the SO_2 production rate reached the desired objective of over 100 l/hr, and maintained about 145 l/hr. Having achieved our primary objective of 100 l/hr, we next explored variations in gas production rate with increasing pressure.

At 12:22 PM a slight back pressure was applied to attain an inlet pressure of 23 psia (1.5 bars). From Figure 3.2 we see that the SO_2 production dropped precipitously because it takes some time to build up pressure in the apparatus until flow resumes. Within a couple of minutes the bayonet once again produced about 140 l/hr of SO_2. This drop in SO_2 production due to incrementing the pressure occurred again at 12:26 PM and 12:30 PM.

As pressure increases, the equilibrium shifts to a lower SO_2 production rate. In Figure 3.2, for clarity the data at pressures of 1, 2, and 3 bars are enclosed in circles. As expected the production rate dropped from 145 l/hr to about 140 l/hr and 135 l/hr, when the pressure increased from 1, 2, and 3 bar respectively. After 12:42 the production rate increased above 150 l/hr, and saturated the instrument at 180 l/hr. This high production rate after 12:42 PM is an artifact of depressurization, which resulted in additional gas released that is not due to acid decomposition.

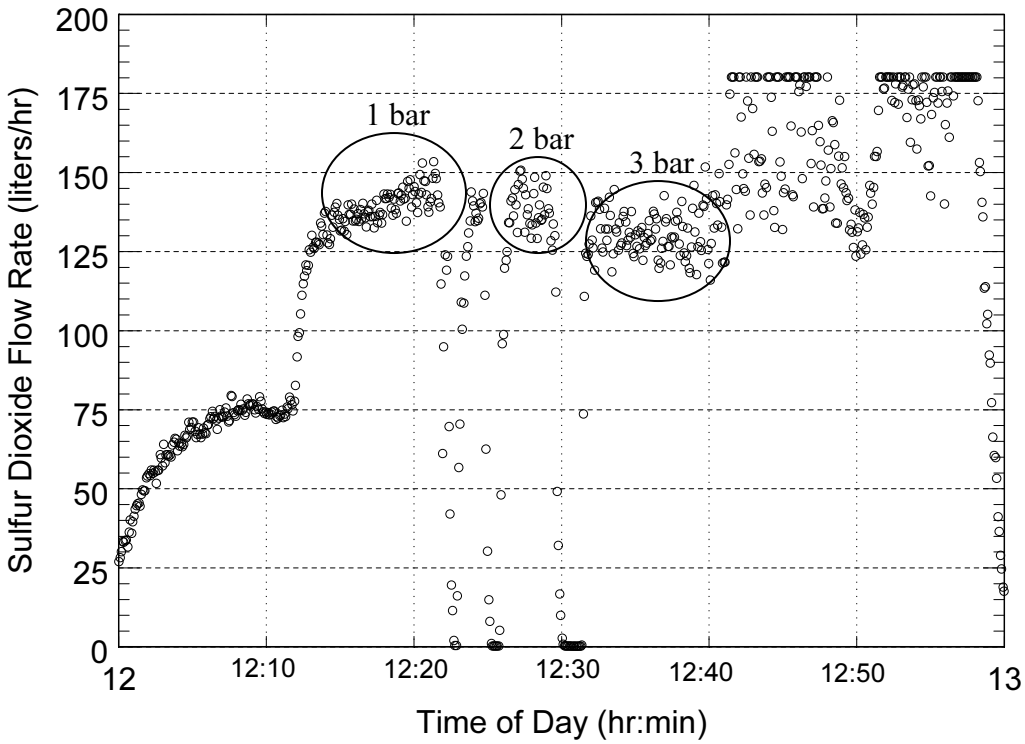

Figure 3.2 SO_2 production rate. The data enclosed by circles at 1, 2, and 3 bar pressure were all taken at an acid inlet flow rate of 7.1 mol/hr.

The gas composition is determined from the oxygen concentration with the assumption that the only other gas is SO_2. The oxygen concentration is plotted in Figure 3.3. Initially, the oxygen concentration overshoots and then settles down to about 34% oxygen, which is in excellent agreement with the expected theoretical value of 33.3% oxygen. To avoid damaging instrumentation during depressurization, the oxygen analyzer was turned off between 12:42 PM and 12:52 PM, and thus no oxygen concentration is plotted in Figure 3.3 during this time interval.

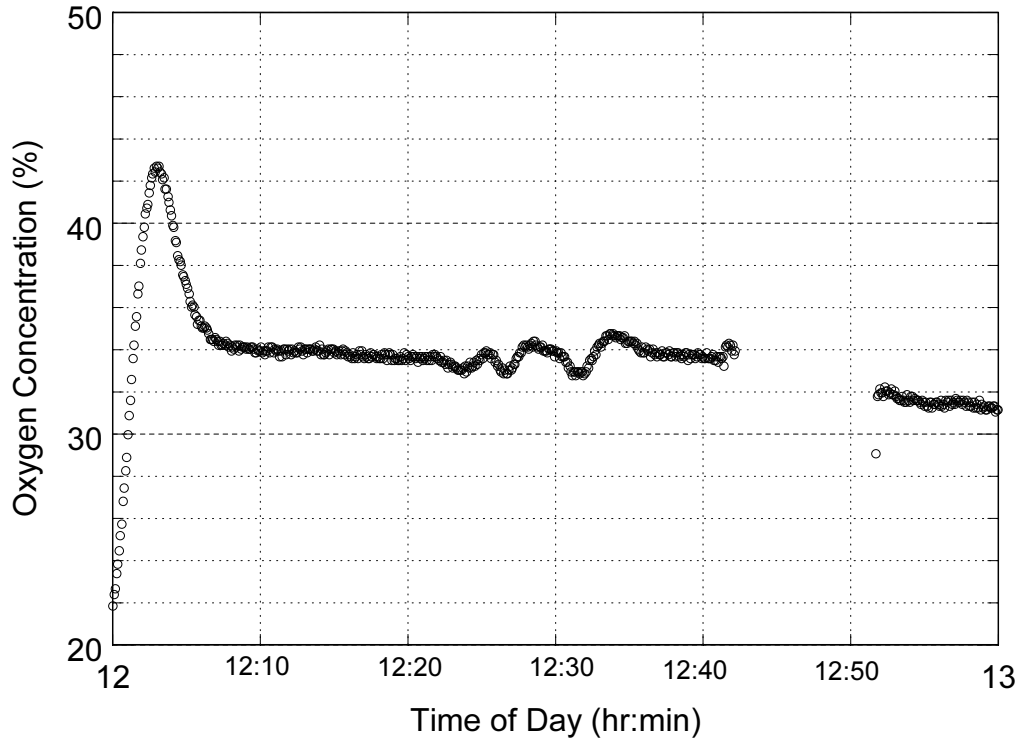

Figure 3.3 Oxygen concentration of produced gas. The experimental value of 34% given in this Figure is in excellent agreement with the theoretical value of 33.3%.

The theoretical maximum acid conversion fraction, F is shown in Figure 3.4 as a function of temperature and pressure. This Figure was obtained using the analysis given in a previous report (Gelbard et al., 2003). From Figure 3.4, the values of F at 850 °C are 0.86, 0.82, and 0.80 for pressures of 1, 2, and 3 bar, respectively. The effluent gas stream was at 30 °C, and the maximum SO_2 production rates at 1, 2, and 3 bar are given by the ideal gas law,

$$\frac{nRTF}{P} = \frac{n\,(0.08205\ \text{liter} * \text{atm}/\text{mol}/\text{K})(303\ \text{K})\,F}{(1\,\text{atm})} \qquad \text{Eq. 1}$$

where n is the acid molar flow rate, R is the ideal gas constant, and T and P are the temperature and pressure, respectively at which the gas flow rate was measured. Thus by substituting the values of n and F into Eq. 1, the theoretical maximum SO_2 production rates are determined and given in Table 3.3 for all four phases of the experiment. The experimentally measured rates from Figure 3.2 are also given in Table 3.3. Notice that the experimental measurements are close to the theoretical maximum production rates. This is based on a catalyst temperature of 850 °C, but the temperature of the catalyst is not measured directly. Instead, we measure the temperature on the outside of the bayonet next to the catalyst, and this temperature is slightly higher than the catalyst temperature. Furthermore, based on our previous tests, the higher the flow rate,

the higher the heat transfer rate required to maintain the catalyst temperature. By using 850 °C as the catalyst temperature the data indicate that there was adequate catalyst and heat transfer to attain near theoretical maximum production of SO_2.

Table 3.3 Comparison of the theoretical maximum and experimental SO_2 production rates

Pressure	Acid flow rate (moles/hr)	Theoretical maximum conversion fraction at 850 °C	Theoretical maximum SO_2 production rate (liters/hour)	Experimental SO_2 production rate (liters/hour)
1 bar	3.6	0.86	77	75
1 bar	7.1	0.86	152	145
2 bar	7.1	0.82	145	140
3 bar	7.1	0.80	141	135

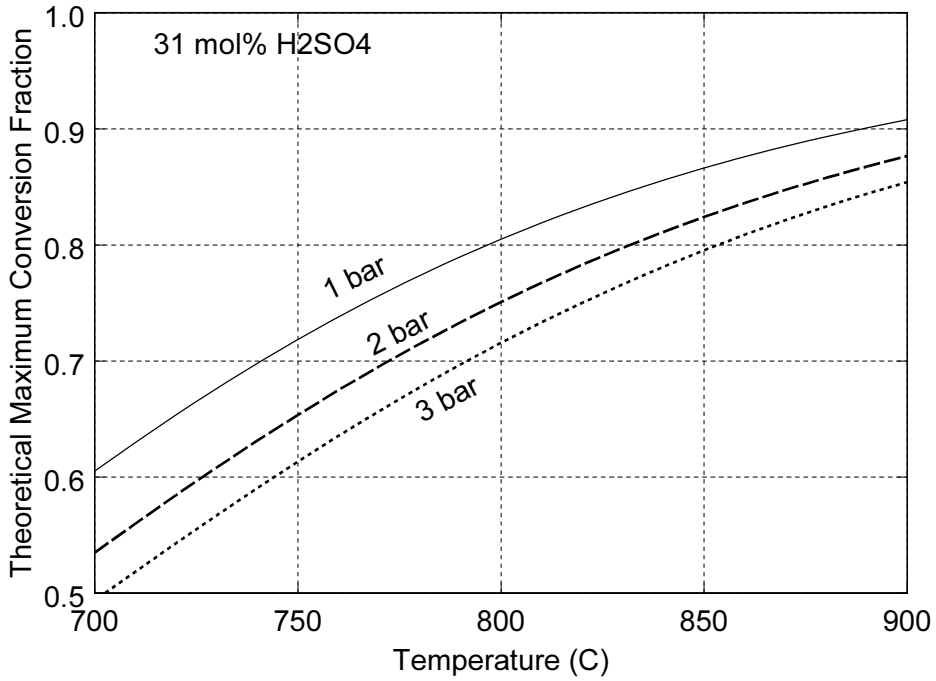

Figure 3.4 Theoretical maximum acid conversion fraction as a function of temperature and pressure (Gelbard et al., 2005).

4 Summary and Conclusions

The initial tests performed on the SNL acid decomposer section confirmed the functionality and performance of the ILS apparatus. The ILS acid decomposer can directly process dilute sulfuric acid (20 mol%), which is to be provided by the Bunsen skid, and produce in excess of 100 liters/hr of SO_2. However, by concentrating the acid to 40 mol%, SO_2 production rates of at least 200 liter/hr can be achieved. SO_2 permeation through Teflon was also addressed to avoid delays or complications during routine maintenance. Earlier tests at SNL identified several needed improvements to the acid decomposer skid, and these were implemented prior to shipment to GA. The primary modifications were a separate additional enclosure within the skid enclosure, and replacement of Teflon tubing with glass-lined steel pipes. The inner enclosure isolates the SO_2 generating components to facilitate maintenance or modification of the skid. The glass-lined steel is more costly and not as readily changed, but provides an almost impervious barrier to SO_2, except for the thin outer edge of the gaskets used between components. The current design has essentially eliminated the problem of SO_2 permeation. Furthermore, in the future many of the gaskets could also be eliminated by having a custom-made glass-lined steel piping network for the apparatus.

Much of the apparatus consists of commercial pumps, tubing, thermocouples, pressure transducers, and structural components. All these worked as expected. The custom-made units are the acid concentrator, the bayonet, and the diagnostic software.

The acid concentrator met the design criteria for the apparatus by concentrating acid from 20 mol% to 40 mol%. We are now investigating modifications to further increase the effectiveness of the unit. The modifications include adding additional acid input ports to increase the initial distribution of acid onto the heated surface of the concentrator. Also, better seals will be installed. Nonetheless, the current unit produced 45 mol% acid from less than 20 mol% acid. The higher the acid concentration going into the bayonet, the more SO_2 can be produced because less heat is used up in vaporizing water.

Pressurized tests confirmed that the ILS bayonet (54 inch long) could produce in excess of 100 liters/hr of SO_2 at 2 bar. At 3 bar, the system produced 135 liters/hr with only 31 mol% acid. The gas production rate was close to the theoretical maximum determined by equilibrium, which indicates that there is adequate catalyst and heat transfer. With higher concentration levels from the concentrator and/or a higher acid flow rate, the bayonet should readily produce more SO_2. In future tests the acid flow concentration and flow rate will be varied to map the parameter space of operations prior to beginning coupled operations with the Bunsen skid.

5 References

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